

## MEMORANDUM

**TO:** Mr. Addison Rice  
Anderson, Mulholland and Associates

**DATE:** June 28, 2016

**FROM:** R. Infante

**FILE:** 1605308CR1

**RE:** Data Validation  
Air samples  
**SDG:** 1605308CR1

### SUMMARY

Full validation was performed on the data for several air samples analyzed for methane by ASTM method D-1946-modified. The samples were collected at the Bristol Myer Squib facility, Humacao, PR site on May 14, 2016 and submitted to Eurofins Air Toxics, Inc. of Folsom, California that analyzed and reported the results under delivery groups (SDG) 1605308CR1.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006; and the QC criteria of the ASTM method D-1946-modified. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use.

### SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
B71A-1D	1605308CR1-02A	05/14/2016	Air	Methane
B71A-3	1605308CR1-04A	05/14/2016	Air	Methane
B71A-4	1605308CR1-05A	05/14/2016	Air	Methane
B71A-5	1605308CR1-06A	05/14/2016	Air	Methane

### REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- o Agreement of analysis conducted with chain of custody (COC) form
- o Holding time and sample preservation
- o Gas chromatography/mass spectrometry (GC/MS) tunes
- o Initial and continuing calibrations
- o Method blanks/trip blanks/field blank
- o Canister cleaning certification criteria

- Surrogate spike recovery
- Internal standard performance and retention times
- Field duplicate results
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- Quantitation limits and sample results

## **DISCUSSION**

### **Agreement of Analysis Conducted with COC Request**

Sample reports corresponded to the analytical request designated on the chain-of-custody.

The Chain of Custody (COC) information for sample B7IA-1D did not match the information on the canister with regard to canister identification. The client was notified of the discrepancy and the information on the canister was used to process and report the sample.

### **Holding Times and Sample Preservation**

All samples analyzed within the recommended method holding time. All summa canisters received in good conditions.

Samples analyzed within method recommended holding time.

### **Initial and Continuing Calibrations**

#### **Methane by ASTM method D-1946 (modified)**

Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.

### **Method Blank/Trip Blank/Field Blank**

Target analytes were not detected in laboratory method blanks.

No trip/field blank analyzed with this data package.

### **Laboratory/Field Duplicate Results**

Laboratory duplicates were analyzed as part of this data set. Target analytes meet the RPD performance criteria of + 25 % for analytes 5 x SQL.

### **LCS/LCSD Results**

#### **Methane**

LCS/LCSD (blank spike) were analyzed by the laboratory associated with this data package. Recoveries and RPD within laboratory control limits.

### Quantitation Limits and Sample Results

Dilutions were not performed (see worksheet).

Calculations were spot checked.

### Certification

The following samples 1605308C-02A; 1605308C-04; 1605308C-05A; and 1605308C-06A were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document.

  
Rafael Infante  
Chemist License 1888





Air Toxics

Client Sample ID: B7IA-1D

Lab ID#: 1605308CR1-02A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	10051713	Date of Collection: 5/14/16 8:12:00 PM
Dil. Factor:	1.66	Date of Analysis: 5/17/16 02:24 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00017	0.00017

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B7IA-3

Lab ID#: 1605308CR1-04A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	10051714	Date of Collection: 5/14/16 8:34:00 PM
Dil. Factor:	1.80	Date of Analysis: 5/17/16 02:46 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00018	0.00016 J

J = Estimated value.

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B7IA-4

Lab ID#: 1605308CR1-05A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	10051715	Date of Collection:	5/14/16 8:57:00 PM
Dil. Factor:	1.62	Date of Analysis:	5/17/16 03:09 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00016	0.00017

Container Type: 6 Liter Summa Canister (100% Certified)



Air Toxics

Client Sample ID: B7IA-5

Lab ID#: 1605308CR1-06A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	10051716	Date of Collection: 5/14/16 8:04:00 PM
Dil. Factor:	1.58	Date of Analysis: 5/17/16 03:33 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00016	0.00019

Container Type: 6 Liter Summa Canister (100% Certified)





eurofins

Air Toxics

## Sample Transportation Notice

Relinquishing signature on this document indicates that sample is being shipped in compliance with all applicable local, State, Federal, national, and international laws, regulations and ordinances of any kind. Air Toxics Limited assumes no liability with respect to the collection, handling or shipping of these samples. Relinquishing signature also indicates agreement to hold harmless, defend, and indemnify Air Toxics Limited against any claim, demand, or action, of any kind, related to the collection, handling, or shipping of samples. D.O.T. Hotline (800) 467-4922

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180 BLUE RAVINE ROAD, SUITE B  
FOLSOM, CA 95630-4719  
(916) 985-1000 FAX (916) 985-1020

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Project Manager Terry Taylor  
Collected by: (Print and Sign) Terry Taylor + David Lindstrand  
Company Anderson Mulholland and Associates Email TTaylor@amhinc.com  
Address 2700 Westchester City Purchase State NY Zip 10571  
Phone 914-251-0400 Fax \_\_\_\_\_

## Project Info:

P.O. # \_\_\_\_\_

Project # \_\_\_\_\_

Project Name \_\_\_\_\_

## Turn Around Time:

☐ Normal☒ Rush1-Day  
specify

## Lab Use Only

Pressurized by:

Date:

Pressurization Gas:

N<sub>2</sub> He

Lab I.D.	Field Sample I.D. (Location)	Can #	Date of Collection	Time of Collection	Analyses Requested	Canister Pressure/Vacuum			
						Initial	Final	Receipt	Final (psi)
01A	B7 IA-1	00300	05/14/16	2011	T0-15	+30	7.5		
02A	B7 IA-1D	00355	05/14/16	2012	T0-15	+30	7.0		
03A	B7 IA-2	N0593	05/14/16	2025	T0-15	+30	13.0		
04A	B7 IA-3	00314	05/14/16	2034	T0-15	+30	10.0		
05A	B7 IA-4	5737	05/14/16	2057	T0-15	+30	6.5		
06A	B7 IA-5	6L1224	05/14/16	2004	T0-15	+30	6.5		
07A	B7 IA-6	00316	05/14/16	2053	T0-15	+30	~47		

Relinquished by: (signature) Date/Time

[Signature] 05/16/16 1500

Received by: (signature) Date/Time

Fed EX

Notes:

Relinquished by: (signature) Date/Time

Received by: (signature) Date/Time

[Signature] EATL 1025 5/17/16

Relinquished by: (signature) Date/Time

Received by: (signature) Date/Time

Lab Use Only

Shipper Name

Air Bill #

Temp (°C)

Condition

Custody Seals Intact?

Work Order #

FedEx

NA

Good

Yes No None

1605308



# DATA REVIEW WORKSHEETS

Project Number: 1605308CR1

Date: 05/14/2016

## REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria from ASTM D-1946 method for measuring permanent gases and light hydrocarbons in refinery and other sources samples using gas chromatography (GC) and a thermal conductivity detector (TCD) and/or flame ionization detection (FID). Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006). The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Eurofins data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: 1605308CR1

Sample matrix: Air

No. of Samples: 4

Trip blank No.: -

Field blank No.: -

Equipment blank No.: -

Field duplicate No.: -

☒ Data Completeness

☒ Laboratory Control Spikes

☒ Holding Times

☒ Field Duplicates

☐ N/A GC/MS Tuning

☒ Calibrations

☐ N/A Internal Standard Performance

☒ Compound Identifications

☒ Blanks

☒ Compound Quantitation

☐ N/A Surrogate Recoveries

☒ Quantitation Limits

☐ N/A Matrix Spike/Matrix Spike Duplicate

Overall Comments: Methane by ASTM method D-1946 (modified)

### Definition of Qualifiers:

J- Estimated results

U- Compound not detected

R- Rejected data

UJ- Estimated nondetect

Reviewer: Rafael Infante

Date: 06/28/2016

## DATA REVIEW WORKSHEETS

## DATA COMPLETENESS

### MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

This image shows a single sheet of white paper with horizontal blue or grey ruling lines. A dashed diagonal line runs across the page from the upper left to the lower right. The paper appears to be part of a notebook or a template for writing.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time. All summa canisters received in good conditions. The Chain of Custody (COC) information for sample B7IA-1D did not match the information on the canister with regard to canister identification. The client was notified of the discrepancy and the information on the canister was used to process and report the sample.				

### Criteria

Aqueous samples – 14 days from sample collection for preserved samples (pH ≤ 2, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C, no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: 4 ± 2 °C): N/A – summa canisters

### Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

All criteria were met N/A  
Criteria were not met see below \_\_\_\_\_

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 01/15/16

Dates of continuing calibration: 05/17/16

Instrument ID numbers: GC-10

Matrix/Level: \_\_\_\_\_ Air/flow \_\_\_\_\_

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.					

#### Criteria

All RFs must be  $> 0.05$  regardless of method requirements for SPCC.

All %RSD must be  $\leq 15\%$  regardless of method requirements for CCC.

All %Ds must be  $\leq 30\%$  regardless of method requirements for CCC.

Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of  $\geq 0.995$  has therefore been utilized as professional judgment.

#### Actions

If any compound has an initial RF or a continuing RF of  $< 0.05$ , estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD  $> 15\%$ , estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD  $> 90\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has a %D  $> 30\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has a %D  $> 30\%$ , estimate positive results (J) and nondetects (UJ).

If any compound has a %D  $> 90\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has  $r < 0.995$ , estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

### Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
All_method_blank_meeth_method_specific_criteria				

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
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No field/trip/equipment blanks analyzed with this data package.

All criteria were met X  
Criteria were not met  
and/or see below

## Blank Actions

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

**Specific actions are as follows:**

If the concentration is  $\geq$  SQL but  $\leq$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is  $\geq$  SQL and  $>$  AL, report the concentration unqualified.

**Notes:**

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

[illegible]

## DATA REVIEW WORKSHEETS

All criteria were met   N/A    
 Criteria were not met  
 and/or see below           

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND	ACTION
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  Surrogate standards not required by the method.  


QC Limits\* (Air)

       LL to UL        to        to        to       

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.



## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below N/A

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

#### 1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ - \_\_\_\_\_ Matrix/Level: \_\_\_\_\_ - \_\_\_\_\_

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
<u>MS/MSD are not required as part of ASTM-method D-1946; blank spike used to assess accuracy</u>					

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
Criteria were not met \_\_\_\_\_  
and/or see below N/A \_\_\_\_\_

#### VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

### MS/MSD – Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ Matrix/Level/Unit: \_\_\_\_\_

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
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[illegible]

**Actions:**

- \* If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).  
\* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

# VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

## 1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?  
 Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
____ LCS/LCSD_(Blank_spike)_analyzed_in_this_data_package;_recoveries_and_RPD_____			
____ within_laboratory_control_limits._____			

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

## 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample ID LCS/LCSD (laboratory duplicate) \_\_\_\_\_

Matrix: Air

Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD  $\pm$  25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
RPD for laboratory duplicate (LCS/LCSD) within laboratory control limits.					

#### Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

## DATA REVIEW WORKSHEETS

All criteria were met N/A  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- \* Area of +40% or -40% of the IS area in the associated calibration standard.
- \* Retention time (RT) within  $\pm 0.06$  seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
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Internal standard not required by the method. Samples quantified by external standard  
method


Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%	IS AREA > + 40%
Positive results	J	J
Nondetected results	R	ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met  
and/or see below       

### XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

1605308CR1-02A

Methane                      RF =

$$[ ] = (44180)/(226379851)$$

$$= 0.000195 \% \text{ OK}$$

All criteria were met   X

**A. Dilution performed**

[illegible]

List samples which have  $\leq 50\%$  solids

[illegible]

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)